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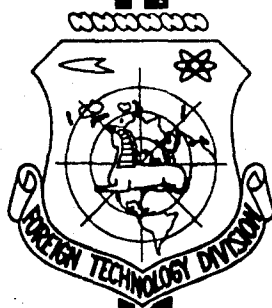
# TRANSLATION

STRUCTURAL STATE AND HEAT  
RESISTANCE OF ALLOYS

By

I. L. Mirkin and V. Z. Tseytlin

## FOREIGN TECHNOLOGY DIVISION



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STRUCTURAL STATE AND HEAT RESISTANCE OF ALLOYS

BY: I. L. Mirkin and V. Z. Tseytlin

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## Structural State and Heat Resistance of Alloys

by

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Z. Tseytlin

Plastic deformation during creep, as well as destruction, appear to be processes, developing gradually in the metal. The rate of these processes depends upon the temperature and applied stress and is determined by the chemical nature (composition) and structure of the substance.

In report [1] have been established the general laws governing the processes of creep and destruction and singular (in type) kinetic equations have been derived

$$\begin{aligned} \tau_{d, \text{destr}} &= \tau_0 \cdot e^{-\frac{U_0 - \gamma \sigma}{RT}}, \\ v_{d, \text{destr}} &= v_0 \cdot e^{-\frac{U_0 - \gamma \sigma}{RT}}, \end{aligned} \quad (a)$$

where  $\tau$  - time prior to destruction of sample;  $\tau_0$  - period of atom oscillations in the lattice;  $U_0$  - energy of atom bonds in the lattice (or heat of sublimation);  $\sigma$  - stress in kg/mm<sup>2</sup>;  $T$  - absolute temperature;  $\gamma$  - stress effectiveness factor.

In both equations, valid, however, only for comparatively greater rates of creep,  $U_0$  and  $\gamma$  appear to be the constants of the substance. This indicates a greater closeness of creep and destruction processes and gives one more basis to affirm, that destruction is a process. The magnitude  $U_0$  is due to chemical nature of the metal, and  $\gamma$  is closely connected with its structure.

S.N. Zhurkov and T.P. Sanfirov showed, that for aluminum at creep tests  $U_0$  does not depend upon the conditions of annealing, but  $\gamma$  does.

At temperatures, approaching the melting points, when diffusion processes are prevalent and a quasiviscous flow of the metal takes place, no doubt the decisive

value for resistance to deformation and destruction is possessed by the chemical nature of the alloy and is signified by its strength of interatomic bonds of the basic solid solution.

However in a temperature zone, relatively sharply removed from the melting point there are border line temperatures for given service conditions ( $\gamma, \delta$ ), the overcoming of which requires a substantial change in chemical nature of the alloys or conversion into another type of crystalline lattice.

And so, in iron-base alloys with volume-centered lattice it is hardly possible to obtain a technically valuable long lasting (1-10 years) resistance to plastic deformation at temperatures, exceeding 900°K (or 630°C), even in the case if the solid solution  $Fe_\alpha(C)$  will be highly alloyed by elements, improving the strength of interatomic bonds. The practice of obtaining alloys confirms this conclusion.

To overcome this temperature boundary it is necessary to change from volume-centered lattice of  $\alpha$ -iron to a more dense face-centered lattice of  $\gamma$ -iron. This is due first of all to the fact that at equal auto-diffusion temperature in a less densely packed  $Fe_\alpha$  lattice the process is approximately twice more intensive than in the  $Fe_\gamma$  lattice.

But above 1000 - 1050°K (or 750°C) the chemical nature of iron-carbon alloys, apparently, cannot assure the necessary long lasting resistance to plastic deformation, and that is why iron-base alloys at any given alloying are still inferior to noniron alloys on the basis of cobalt, nickel, chromium, molybdenum etc.

At temperatures, removed from the melting point <sup>and</sup> not reaching "boundary" temperatures the basic structural state of the alloy is of particular great importance. For components of electric power installations, working for a long time at medium temperatures (tens and hundreds of thousands of hours, i.e. years and decades), together with the initial structural state of greater importance is the kinetic factor determining the ability of the alloy to retain for a long time the initial structural state, assuring high heat resistance ("structural stability" of the alloy).

Alloys with sluggish diffusion processes, coagulation processes and volumetric reactions between the phase components (which causes a small rate of structural and phase conversions) retain heat resistance for a much longer period of time, creep and relaxation take place at lower rate.

The initial structural state of the metal is determined by the composition and state of mother (starting) solid solution and separation phases and also by the mutual disposition and interaction of these phases.

Heat resistance of basic solid solution appears to be a function of its chemical composition, i.e. concentration of various alloying elements in it.

The elements, introduced into the solid solution, affect it in various ways: some do not promote a rise in heat resistance, and others even reduce same. For example, introduction of 20% Fe does not raise the heat resistance of solid nickel alloys.

Heat resistance of the solution can be increased only upon its alloying with elements, raising the bond forces, inhibiting diffusion and especially auto-diffusion processes and raising the recrystallization temperature and resistance to shear etc. It was established, that of many elements only tungsten slows down sharply auto-diffusion of nickel.

The presence of several elements (e.g. Mo, W, Cr, Co and others) is more effective than the introduction of an equal in atomic concentration amount of one element. The diffusion mobility changes non-proportional to the concentration, and different components give a nonuniform effect.

For example, it was established, that if nickelchrome alloys (with 15% Cr) are introduced to 10% Co, W, Mo and to 2% Nb the heat resistance of the alloys will increase.

The resistance to creep was increased and, which is of special importance, the relaxation stability. It was confirmed, that simultaneous introduction into the alloy of several alloying elements has exerted a more favorable effect on the heat resistance. And so, during additional introduction of 2% Nb in an alloy containing 5% of

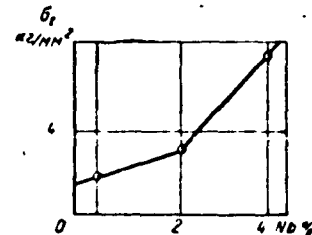
Mo and 5% of W, has considerably increased the long lasting strength and resistance to relaxation at 700°.

But solid solutions, even poly-component ones, alloyed with the mentioned elements in amounts, close to the limit of their solubility, possess relatively low heat resistance.

The presence of excessive phases in the skeletal or finely dispersed form or separation of these phases during early stages of aging (after technological annealing) appears to be an ordinary condition for the creation of high resistance to creep and relaxation.

1. Alloying of nickelchrome alloys with molybdenum, tungsten and niobium in amounts far from the limit of solubility, does not give a technically suitable heat resistance. An increase in the niobium content to the amount, exceeding its solubility limit in such alloys (4% Nb), led to the formation of niobium-containing excess phases and sharply raised the heat resistance (fig.1). At equal conditions the remaining stress ( $\sigma_t$ ) after relaxation testing in a heterogeneous alloy was by 100% higher, than in single phase.

2. Nickelchrome alloys, in which Al and Ti are included, form strengthening separation phases. The oversaturated solid solution (after hardening) in the pro-



cess of testing to the beginning of separ. Fig.1. Change in value of remaining stress after 1400 hrs of testing for relaxation at 700° during an increase in niobium content, 15 kg/mm², 5% Mo and W, 3% Fe. at 700° during an increase in niobium content, 15 kg/mm², 5% Mo and W, 3% Fe.

considerably lower heat resistance than after their separation. At the beginning the rate of deformation of the hardened alloy is approximately 3 times greater than of the annealed one. But, later on, in ratio to the separation of reinforcing phases, the resistance of the hardened alloy rise sharply (fig.2) and the rates of deformation become equal within a period of 10000 hrs.



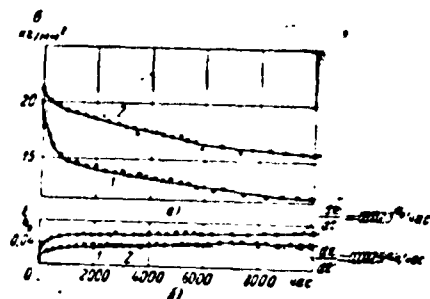


Fig.2. Relaxation curves (a) and change in plastic deformation (b) of Ni-Cr alloy in heterogeneous alloys and steel or in with 2% Al and 2% Ti at  $660^{\circ}\text{C}$ ,  $25\text{ kg/mm}^2$  1-hardening; 2-hardening and annealing. supersaturated solid solutions, from which at high temperatures are separated excessive phases. In fig.3 is shown a rise in time to the point of destruction during change over from solid solution Fe(Ni,Cr) to a heterogeneous alloy followed by a rise in the amount of excessive phase.

For heterogeneous alloys (or for the heterogeneous state of supersaturated alloys) of great importance is also the structural state of the mother solid solution, produced by thermal treatment and plastic deformation.

To slow down phase conversions it is important to reduce the rate of diffusion in the alloy. This is particularly important for alloys, working under conditions of creep and relaxation, because it was experimentally shown by M. Koen as well as by I Ya. Degtyar and V.S. Mikhailenkov, that plastic deformation speeds up diffusion proportionally to the rate of deformation.

The chemical and structural heterogeneity of the solid solution may considerably decrease the resistance to plastic deformation. It is known, that the presence in initial state in the structure

of chromickel austenitic steel (18 Cr-8 Ni) of ferrite reduces heat resistance. The use of such steel in the role of heat

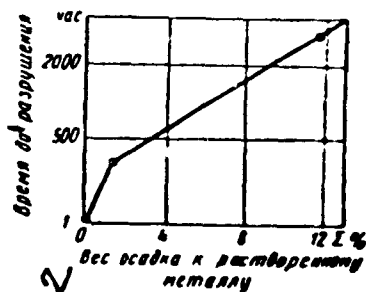


Fig.3. Change in time to destruction of alloy with 16% Cr and 32% Ni (at variable content of Mo and W) depending upon the increase in weight of electrolytically separated deposition. Phases of separation of  $\text{Ni}_2\text{Cr}$  and  $\text{FeNi}_2\text{C}$ ,  $700^{\circ}\text{C}$ ,  $12\text{ kg/mm}^2$ . 1-time to destruction; 2-weight of deposition to dissolved metal.

resistant is stipulated by technological requirements (weldability). It was also established that in steel with uniform basic structure (14 Cr-14 Ni) is possible the appearance of a ferromagnetic alpha-phase [2]-[4] as result of concentrational changes during carbide formation. Such separation of alpha-phase has a negative effect on the long lasting and cyclic strength, as well [4] as on the relaxation stability (fig.4).

At other conditions being equal, with the increase in grain dimension in the initial structure the long lasting heat resistance of the alloy at sufficiently high temperatures and low stresses, increases. This is due to two causes.

1. Hypothesis about the plasticity diffusion mechanism, realizable through the boundaries of grains, was confirmed by [5], [6] and others. Metallographically determined was the fraction of deformation on account of the boundaries as compared with the deformation along the grain during creep of aluminum [7]. At low temperatures (less than 200°) deformation goes almost exclusively by the grain and the fraction of displacement over the boundaries constitutes less than 10%. But when testing at above 250° deformation over the boundaries constitutes more than 85% of total deformation. Consequently, it is only natural, that with the decrease in summary extension of the boundaries the possibility of growth of plastic deformation by the displacement of atoms in the boundary zone decreases.

2. If the initial structure consists of fine grains, then at sufficiently high temperatures the grains becomes strengthened and creep is eased.

A much higher heat resistance of large grain steel and alloys [8] - [11] especially at high temperatures, was confirmed experimentally.

That is why only one example is given [8]

- effect of grain dimension on the resistance to relaxation of carbon steel. (fig. 5).

Not only the summary extent of boundaries, but also their nature and state have

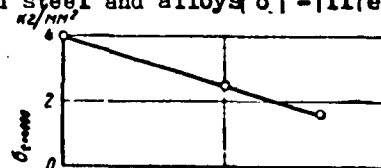


Fig. 4. Change in relaxation stability of austenitic type 14 Cr-14 Ni steel (magnetic phase) after 4000 hrs of testing at 650° in relation to the amount of alpha phase. 1-amount of ferromagnetic phase.

an effect on the resistance to plastic deformation in the process of testing and on the service of the alloy at high temperatures. It can be stated [6] that the lower the disruptions in the correctness of intergrain lattice, the stronger are the interatomic bonds of that lattice, the higher is the resistance of plastic deformation boundaries, heat resistance and especially the plasticity of the alloy. The development of new methods of obtaining metals and alloys of special purity by distillation, zonal smelting allows to investigate the effect of even small admixtures on the state of grain boundaries and on their resistance to plastic deformation.

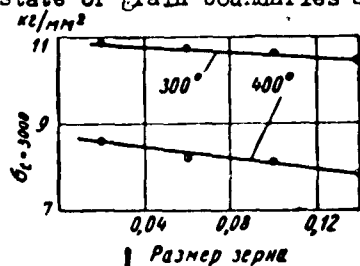


Fig. 5. Effect of grain dimension on residual stress after 3000 hrs of testing for relaxation. 1-grain dimension.

The level of heat resistance applicable for technology can be attained in alloys with heterogeneous structure, i.e. in the presence of the structure of reinforcing excessive phases.

By the diffusion equation was calculated [12] that in the lattice of nonalloyed ferrite at room temperature is perfected an insignificantly small number of jump overs of carbon atoms (2 per sec). At 200° it becomes noticeable ( $3 \times 10^5$  per sec), at 400° - considerable ( $3 \times 10^8$ ), and at 600° - mass scale ( $1 \times 10^{10}$ ).

At room temperature the ferrite-perlite structure of the steel has practically unlimited stability in time: already at 400° it may coagulate at noticeable rate and consequently to use steel at above that temperature it is obligatory to alloy the ferrite to slow down carbon diffusion. For operation of the steel at 500-600° it not only requires complex alloying of the ferrite, but also the creation of special carbides for sharp stabilization of the structure. Finally, above 620-650° it is necessary to change the type of alloy lattice, i.e. to change to austenitic steel.

Of decisive importance is also the chemical nature of the separation phases, determining the strength of bonds of their crystalline lattice. For such phases there are also boundary temperatures, the surmounting of which is required to assure high heat resistance a change in chemical nature and type of crystalline lattice of

separation phases.

In materials, designated for operation at 550-570°, the basic reinforcing phase appears to be alloying cementite. Of second grade importance are special carbides (VC and generally  $MeC$ ). When working at above these temperatures iron-base carbides do not assure high heat resistance of the steel. From 570 - 580° approximately to 700° the basic one appears to be chromium carbide. In a relatively narrow temperature zone from 580 to 600 - 610° the basic separation phase appears to be trigonal chromium carbide  $Me_7C_3$ , and at 600-650° - cubical  $Me_{23}C_6$  carbide.

Above 700-750° carbide reinforcing phases become inferior to intermetallide compounds, having different relationships with the mother solid solution, which even at this temperature zone must be selected principally different (of noniron base).

For example, at 700-900° in nickel-chromium base alloys the main reinforcing agent appears to be the  $Ni_3(Al, Ti)$  type phase.

In the mentioned temperature zones the change over from much lower temperatures to much higher ones requires complication of phase separation composition. And so, in the first zone at temperatures of less than 300-350° the reinforcing phase can be unalloyed cementite (cementite phase of variable composition may substantially change the content of carbon, it has a broad zone of homogeneity). At much higher temperatures the diffusion mobility of iron atoms and carbon atoms becomes already so much greater, that: 1) it considerably accelerates the processes of carbide coagulation and transition from plastic form to "point" form (granular), and then also to globular and 2) with the increase in serviceability it causes dissociation of the cementite with the formation of structurally free graphite, as result of which there is a sharp drop in the heat resistance of the steel.

An increase in working temperature to 400-450° requires the introduction of Cr or Mo, which not only are included in the solid solution, strengthening it and raising its heat resistance, but are also capable of replacing a part of the iron atoms in the cementite.

They increase the strength of interatomic bonds and hamper diffusion processes, leading to accelerated coagulation of carbide, and sometimes even to decomposition of same with the formation of structurally free carbide. Simultaneous introduction of the above mentioned elements is much more effective. It is known, for example, that the presence of one molybdenum in cementite of tubular steel type 15M is in no position to prevent the appearance, at a temperature of about 500° at lasting heating, of structurally free carbide, which in particular led to serious emergency of steam pipe line at the electric power station at Spring-Dale (USA).

The presence in steel of one molybdenum indicates that in the process of long lasting service the molybdenum is displaced from the ferrite into carbides and the heat resistance of the steel is reduced. It was established [13] that after exploitation for a period of 15-25 thousand hours at 500° pipes made of steel 15M from one fourth to one half of the amount of molybdenum contained in the ferrite, has transformed into separation phase. As result of this the heat resistance of the metal of such pipes decreases sharply (fig.6). Therefore a further increase in the working temperature requires an even greater complication of the cementite or carbide content on account of introducing into it atoms of vanadium and other elements. At temperatures, close to boundary temperatures for steel with volumetrically-centered cubical lattice are introduced already such amounts of above mentioned elements, that in addition to the  $M_3C$  phase are also formed  $MeC$  type carbides, necessary for complicating the phase composition, inhibition of volumetric reactions between basic phases and for retention of alloyability of solid solution.

It was established, that for perlite class steel ( e.g. chromium-molybdenum-vanadium, chromium-molybdenum ) the formation of a ferrite-carbide mixture without preliminary martensite conversion produces much higher heat resistance.

This is explained by the fact [14] that during point and granular structure of perlite, which may originate in steel as result of decomposition of martensite, there is a greater number of nuclei of simultaneous origination and development of plastic

deformation at lower resistance as compared with the lamellar structure.

Highest heat resistance at 450 - 570° is possessed by perlite steel with carbide ferrite mixture, formed after the annealing of products of bainite reaction [15], - [17]

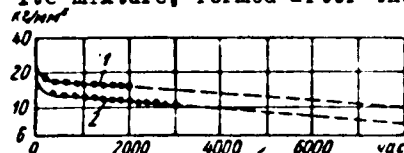


Fig. 7. Relaxation curves of 25KHFA steel at 500°C, 25 kg/mm². 1-normalization at 920° + annealing 650°. 2 hours; 2-hardening 920° + annealing 650°, 2 hours.

It was established that Cr-Mo-V-steel, annealed after normalization, possesses consider-

ably greater heat resistance than the very

same steel, annealed under analogous conditions after hardening [17]. This is evident if only by comparing the stress

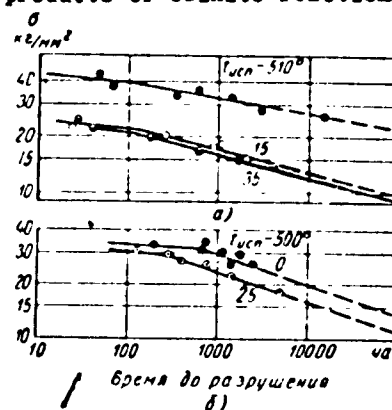


Fig. 6. Curves "logarithm of time up to destruction - logarithms of destructive stress" of tubular steel 15M. a-in initial state (1500 hrs); b-after long operation (25000 hrs). 1-time till destruction.

reduction curves in the process of relaxation of improved steel and of steel, subjected

to normalization with annealing (fig. 7). This is explained by a more favorable distribution of alloying elements between the solid solution and carbide phase. In chromemolybdenum steel 25KHFA after normalization with annealing [18] the summary weight of the deposition after electrolytic solution (5.55% of the weight of dissolved

metal) is greater, than after hardening with very same annealing (4.39%). But this takes place on account of the iron at a uniform content in the carbide phase of Cr, Mo, V ( $\sum_{Cr, Mo, V} \approx 0.7\%$ ).

In this way, the concentration of these elements in solid solution after normalizing with annealing is higher, than after improving, i.e.

$$\left( \frac{Mo + Cr + V}{Fe} \right)_{\text{solid solution after normalizing}} > \left( \frac{Mo + Cr + V}{Fe} \right)_{\text{solid solution after improving.}}$$

At moderate temperatures the advantages of lamellar and finely-lamellar form of perlite are beyond any doubt. At boundary temperatures there can be instances, where the initial advantage of lamellar structure becomes exhausted with the expiration of time as result of higher rate of crushing the plates and coagulation.

In the case of 15M steel at 500° for a period of 15000 hrs at initial lamellar structure the limit of long lasting strength dropped from 25 to 11 kg/mm<sup>2</sup>, and at an initial granularity - from 15 to 13 kg/mm<sup>2</sup> [13].

Just as for alloys, having polymorphous conversions, with a rise in temperature it is necessary to complicate not only the composition of the mother solid solution, but also the reinforcing phases of alloys, having no polymorphous conversions. At a working temperature of up to 600° the basic reinforcing phase in such steel may serve the unalloyed cubical chromium carbide Cr<sub>23</sub>C<sub>6</sub>. But a further rise in temperature to 650° and over requires additional alloying with elements, increasing the bond forces not only of the die, but also the carbide phase, which transforms already into solid solution several metals on cubical chromium carbide Fe<sub>23</sub>C<sub>6</sub>-base. Complication of the carbide phase composition with molybdenum and tungsten raises its thermal stability and promotes a general rise in the heat resistance of the alloy. This pertains also to intermetallide phase. No doubt, that even a small solution of chromium, molybdenum, tungsten in the intermetallide phase of the Ni<sub>3</sub>Al type, it promotes a rise in its thermal stability, since it hampers the occurrence of the diffusion processes, leading to the formation and growth of formations, which is indicated by the analysis with the aid of the fluctuations theory.

The original structural state does in many respects promote the heat resistance of alloys. But at long lasting operation of the metal at high temperatures of importance is the stability of the structural state for a period of tens of thousands of hours. Intensive occurrence of the physico-chemical processes (diffusion, interchange), leading to quantitative and qualitative changes in phases and coagulation of

crystallites of separation phases, causes an acceleration in plastic deformation. For example, in 12KH12V4M steel in the process of gain without stress at 600° after 3000 hours in addition to  $\text{Me}_{23}\text{C}_6$  carbide there is also the Laves phase  $\text{M}_\text{A} (\text{M}_\text{V})_2$  which after 5000 hours of aging becomes predominant [19].

Long lasting tests at 600° of an alloy containing 20% Ni, 20% Co [20] is characterized by a discontinuity in plastic deformation growth curves in the process of

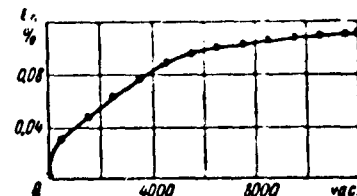


Fig. 8. Change in plastic deformation in relaxation (5000-6000 hrs), after which relation to the testing time for relaxation at 660°, 25 kg/mm².

the rate of growth of plastic deformation at further tests decreases (fig. 8). An analogous discontinuation has been detected on curves

indicating changes in hardness, impact strength, magnetic permeability. A study of the kinetics of change in phase composition showed, that after 5000 hrs the rate of  $\text{Me}_{23}\text{C}_6$  carbide separation from solid solution is considerably lower (fig. 9). This, most likely explains the presence of a discontinuity on the property change curves of the alloy. At more intensive processes, leading to phase changes, the properties of the alloy, and especially, its resistance to deformation change intensively. After

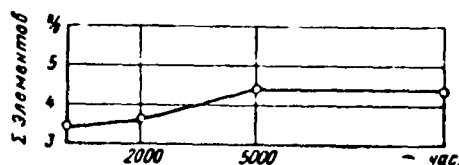


Fig. 9. Change in summary content of alloying elements in carbide phase of the alloy in the process of isothermal exposure at 660°.

this, the phys-chem. processes become more sluggish, further intensification in deformation and in the change of other properties is slowed down sharply.

Of greater importance for long working heat resistant alloys is the kinetics of

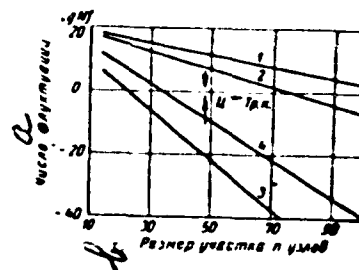
phase conversion processes and, especially, the kinetics of separation and regeneration of excessive phases.

To slow down phase conversions it is important to reduce the rate of diffusion, particularly in alloys, working under conditions of creep and relaxation. However together with the outstanding role of the rate of diffusion processes, very important



in many instances even decisive, is the factor of probability of concentrational fluctuations. The fluctuation theory explains the repeated inhibition of the phase separation process during the complication of the composition of the alloy and increase in the differences between the composition of the separating and mother phases [21]. For example, separation of chromium cementite instead of ordinary unalloyed one reduces the number of carbide nuclei by 2-4 magnitudes (fig.10, 1 and 2) and the transition into trigonal carbide reduces the number of nuclei by several magnitudes (fig.10,3).

Separation of metastable and intermediate phases (particularly coherently bound with mother solid solution) and their subsequent regeneration with the expiration of time are also to a large extent predetermined by the probability of formation of fluctuations of critical dimension. And so for example, separation of cementite type carbide in the role of intermediate phase is preceded by the separation of trigonal carbide largely because the probability of formation of the first one is greater by many magnitudes (see arrows in fig.10).



Under definite conditions (fig.11)

for the formation of new phase nuclei are important not only components, enriching the new phase (Ti in nickel alloys) but

Fig.10. Comparing number and dimensions of fluctuation nuclei of carbides of various type in chrome steel:

1-Ni ( $\text{Fe}_3\text{C}$ ); 2- steel with 2% Cr. Ni ( $\text{Fe,Cr}$ )<sub>3</sub> C in carbide 15% Cr; 3- steel with 2% Cr, Ni ( $\text{Cr,Fe}$ )<sub>7</sub>C<sub>3</sub> in carbide 40% Cr; 4- steel with 7% Cr, Ni ( $\text{Cr,Fe}$ )<sub>7</sub>C<sub>3</sub>. a-number of fluctuations; b- dimension of section n of units.

also the components expelled from the volume of the nucleus (Cr).

An analysis made enabled to reveal a case, where during additional alloying of the alloy the rate of separation of new phase is slowed down on account of reducing the probability of necessary concentrational fluctuation by many magnitudes. And so for 14 Cr - 14 Ni + W type steel tested within a period of 9000 hrs at 590° it is possible to prove the following:

weight of carbide deposition, formed by the method of anodic solution in % to weight of dissolved metal... 1.3

Average diameter of carbide particles (by measuring under an electronic microscope) in  $\mu\text{m}$ ...  $1 \cdot 10^{-5}$

number of carbides per  $1 \text{ cm}^3$ ...  $3 \cdot 10^{13}$

average rate of nucleation of  $\text{Fe}_2\text{C}_6$  carbides per sec...  $1 \cdot 10^6$

ditto, calculated by fluctuation theory...  $2 \cdot 10^6$

rate of growth of carbides in  $\text{cm/sec}$ ...  $5 \cdot 10^{-13}$

In this way, to form each atomic layer several hours of exposure are necessary.

In simple carbon steel the rate of perlite grain growth is greater by  $10^9$  times than the rate of growth of carbides in above mentioned alloyed steel.

The above stated shows, that of much greater importance for heat resistant long service alloys is the structure and especially the kinetics of lasting changes in structural state.

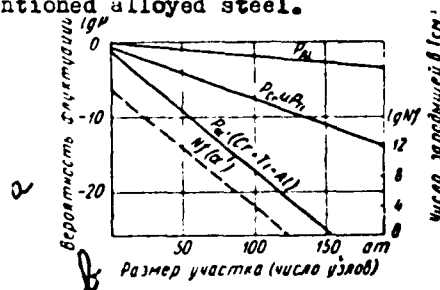


Fig. 11 The probability, number and dimensions of fluctuation nuclei of  $\alpha'$ -phase in manganic type alloy; 1-probability of fluctuation; 2-dimension of section (number of units); 3-number of nuclei in  $1 \text{ cm}^3$

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